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REPULIEK VAN SUID AFRIKA



Certificate

REPUBLIC OF SOUTH AFRICA

PATENT KANTOOR
DEPARTEMENT VAN HANDEL
EN NYWERHEID

PATENT OFFICE
DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

- 1) Scuth African Patent Application No. **2003/2621** accompanied by a Provisional specification was filed at the South African Patent Office on **3 April 2003** in the name of **Merisol (RSA) (Pty) Limited** in respect of an invention entitled: "**Purification of phenols**"

- 2) The photocopy attached hereto is a true copy of the provisional specification and drawing filed with South African Patent Application No. **2003/2621**.

Geteken te

PRETORIA

Signed at

in die Republiek van Suid-Afrika, hierdie

in the Republic of South Africa, this

5th

dag van

April 2004

day of

A handwritten signature in blue ink, appearing to read "B Goepa".

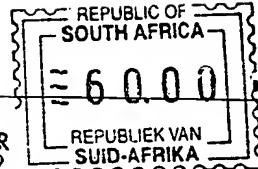
Registrar of Patents

| REPUBLIC OF SOUTH AFRICA | | | | PATENTS ACT, 1978 | |
|--|--|----------------------------|----|-------------------|-----------------|
| REGISTER OF PATENTS | | | | | |
| OFFICIAL APPLICATION NO. | | LODGING DATE : PROVISIONAL | | ACCEPTANCE DATE | |
| 21 | 01 | 2003/2621 | 22 | 26 May 2003 | 43 |
| INTERNATIONAL CLASSIFICATION | | LODGING DATE : COMPLETE | | GRANTED DATE | |
| 51 | | 23 | | | |
| FULL NAME(S) OF APPLICANT(S) / PATENTEE(S) | | | | | |
| 71 | MERISOL (RSA) (PTY) LIMITED | | | | |
| APPLICANTS SUBSTITUTED : | | | | | DATE REGISTERED |
| 71 | | | | | |
| ASSIGNEE(S) | | | | | DATE REGISTERED |
| 71 | | | | | |
| FULL NAME(S) OF INVENTOR(S) | | | | | |
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| PRIORITY CLAIMED | | COUNTRY | | NUMBER | DATE |
| N.B. Use international abbreviation for country. (See Schedule 4) | | 33 | | 31 | 32 |
| TITLE OF INVENTION | | | | | |
| 54 | PURIFICATION OF PHENOLS | | | | |
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| PATENT OF ADDITION NO. | | DATE OF ANY CHANGE | | | |
| 61 | | | | | |
| FRESH APPLICATION BASED ON | | DATE OF ANY CHANGE | | | |

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

APPLICATION FOR A PATENT AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) - Regulation 22)

The grant of a patent is hereby requested by the undermentioned applicant
on the basis of the present application filed in duplicate.



Form P.1

| | |
|-------------------------|------------|
| OFFICIAL APPLICATION NO | |
| 21 | 02003/2621 |

| | |
|---------------|--|
| DMK REFERENCE | |
| P26466ZA00 | |

| | |
|------------------------------|-----------------------------|
| FULL NAME(S) OF APPLICANT(S) | |
| 71 | MERISOL (RSA) (PTY) LIMITED |

| | |
|-----------------------------|--|
| ADDRESS(ES) OF APPLICANT(S) | |
| | 2 Sturdee Avenue Rosebank Johannesburg South Africa |

| | |
|--------------------|--|
| TITLE OF INVENTION | |
| 54 | PURIFICATION PHENOLS |
| | THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P2 The earliest priority claimed is |
| | THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO. 21 01 |
| | THIS APPLICATION IS FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON APPLICATION NO. 21 01 |

| | |
|---|--|
| THIS APPLICATION IS ACCCOMPANIED BY : | |
| <input checked="" type="checkbox"/> | 1a A single copy of a provisional specification of 18 pages. |
| <input type="checkbox"/> | 1b Two copies of a complete specification of _____ pages. |
| <input type="checkbox"/> | 2a Informal drawings of _____ sheets. |
| <input checked="" type="checkbox"/> | 2b Formal drawings of 1 sheets. |
| <input type="checkbox"/> | 3 Publication particulars and abstract (form P8 in duplicate). |
| <input type="checkbox"/> | 4 A copy of figure. _____ of the drawings for the abstract. |
| <input type="checkbox"/> | 5 Assignment of invention (from the inventors) or other evidence of title. |
| <input type="checkbox"/> | 6 Certified priority document(s). |
| <input type="checkbox"/> | 7 Translation of priority document(s). |
| <input type="checkbox"/> | 8 Assignment of priority rights. |
| <input type="checkbox"/> | 9 A copy of form P2 and a specification of S.A. Patent Application. |
| <input type="checkbox"/> | 10 A declaration and power of attorney on form P3. |
| <input type="checkbox"/> | 11 Request for ante-dating on form P4. |
| <input type="checkbox"/> | 12 Request for classification on form P9. |
| <input type="checkbox"/> | 13a Request for delay of acceptance on form P4. |
| <input type="checkbox"/> | 13b |
| 21 01 | |

DATED

3 April 2003

| | |
|---------------------|---|
| ADDRESS FOR SERVICE | |
| 74 | D M Kisch Inc Inanda Greens Business Park 54 Wierda Road West Wierda Valley SANDTON |

The duplicate will be returned to the applicant's address for service as

| | |
|---|--|
| Patent Attorney for Applicant(s) | |
| | REGISTRAR OF PATENTS DESIGNS, TRADE MARKS AND COPYRIGHT |
| OFFICIAL DATE STAMP 2003 -04- 03 | |
| REGISTRAR OF PATENTS | |
| REGISTRATEUR VAN PATENTE, MODELE, HANDELSMERKEN EN OUDTEURSREG | |

REPUBLIC OF SOUTH AFRICA

PATENTS ACT, 1978

PROVISIONAL SPECIFICATION
(Section 30 (1) - Regulation 27)

| | | | | | |
|------------------------------|--|--------------|----|---------------|------------|
| OFFICIAL APPLICATION NO. | | LODGING DATE | | DMK REFERENCE | |
| 21 | 01 | 2003/2621 | 22 | 3 April 2003 | P26466ZA00 |
| FULL NAME(S) OF APPLICANT(S) | | | | | |
| 71 | MERISOL (RSA) (PTY) LIMITED | | | | |
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| 72 | LUND, Grant James COOGAN, Edward John JANSEN, Wilhelmina | | | | |
| TITLE OF INVENTION | | | | | |
| 54 | PURIFICATION OF PHENOLS | | | | |

This invention relates to the purification of phenols in a feedstock containing
5 phenols and neutral oils and/or tar bases.

BACKGROUND ART

Feedstocks containing mixtures of phenols (such as phenol, cresols,
10 ethylphenols, xylenols and C3 phenols) and neutral oils and/or tar bases are obtained from various sources such as petroleum and coal processing facilities.

The separation of phenols from tar bases and especially, neutral oils have always posed a challenge since certain neutral oils have boiling points close to
15 that of the phenols and due to the fact that azeotropes are formed between the neutral oils and phenols. Normal distillation processes are accordingly not suitable to separate phenols from neutral oils and/or tar bases to the required purity.

20 Liquid-liquid extraction is a known method of separating phenolics from neutral oils and/or tar bases. The prior art discloses that both single solvent and dual solvent processes have been employed to achieve such a separation. Dual solvent processes are preferred. In these processes a solvent is used to extract the phenolics and a counter-solvent is used to
25 extract the neutral oils. The solvent and counter-solvent are selected to be

immiscible and after extraction the solvent and counter-solvent layers are separated and the phenolics are recovered from the solvent layer while the neutral oils are recovered from the counter-solvent layer.

- 5 The solvent usually is a polar solvent and the counter-solvent usually is a non-polar solvent. The counter-solvent often comprises paraffins such as hexane, heptane, petroleum ether, octane and diesel. The polar solvent often comprises an aqueous solution of methanol, ammonia, acetamide, acetic acid, ethanol and glycols. In this regard see for example US Patent
- 10 2,666,796 where aqueous methanol and hexane were used as solvent and counter-solvent respectively. In the Journal of Applied Chemistry, 21 June 1952, page 314, and the Journal of Applied Chemistry, 3, March, 1953, page 98, aqueous triethylene glycol was used as solvent. In Fuel processing Technology, 28 (1991) page 287 aqueous solutions of ethylene glycol, diethylene glycol and triethylene glycol were used as solvents. In South African patents 98/11312 and 2001/9287 aqueous solutions of glycols were used as solvents and they were used in combination with counter-solvents.
- 15
- 20 In cases where the solvent comprises a mixture of water and a solvent compound (e.g. glycols) a solvent layer is obtained which contains the water, the solvent compound (e.g. glycols), phenolics, some neutral oils and/or tar bases and some counter-solvent. The first step is usually to

subject the mixture to a water recovery distillation process to strip the water from this mixture. The bottoms product consists mainly of the solvent compound (e.g. glycols) and phenolics, which bottoms product is then subjected to distillation to separate the phenolics from the glycols.

5

In South African patent 2001/9287 the solvent comprised a mixture of glycol and water and the water recovery distillation process was carried out under vacuum to prevent thermal decomposition of the glycol. The overheads product of the water recovery distillation process (consisting of
10 water, some counter-solvent, some phenols and some neutral oils and/or tar bases) was condensed and was then mixed with counter-solvent to extract the condensate with the counter-solvent. A decanter was then used to achieve separation of the aqueous and organic layers which formed. It was suggested that the counter-solvent added resulted in extraction of organic
15 material into the counter-solvent layer and it also resulted in better phase separation in the decanter.

As stated above, after or during the liquid-liquid extraction step the immiscible solvent and counter-solvent layers are separated from each other
20 and the phenols are recovered from the solvent layer (as described above) while the neutral oils and/or nitrogen bases are recovered from the counter-solvent layer.

In South African patents 98/11312 and 2001/9287 the counter-solvent layer was subjected to a counter-solvent recovery distillation process to yield recovered counter-solvent (as an overheads product) and neutral oils and/or tar bases (as a bottoms product). The recovered counter-solvent

5 was then recycled to the liquid-liquid extraction step and the neutral oils and/or tar bases were then routed to a neutral oils and/or tar bases processing facility. South African patent 98/4165 describes a similar process for recovering the counter-solvent and in that case the neutral oil (as bottoms product) was either used as a feedstock for napthalenes or it

10 was combusted for fuel value.

It has now been established that to obtain optimum conditions for optimum separation of phenols from neutral oils during the liquid-liquid extraction the ratio of solvent to counter-solvent is such that some of the solvent is

15 transferred to the counter-solvent layer. During the counter-solvent recovery distillation process the solvent forms part of the bottoms product with the neutral oils and/or tar bases. Since the bottoms product was in the past forwarded to a neutral oils and/or tar base processing facility, or was used as a napthalenes feedstock, or was combusted as fuel, the solvent

20 contained therein was lost to the process.

It is an object of the present invention to reduce the above disadvantage.

DISCLOSURE OF THE INVENTION

According to the present invention a process for purifying phenols from a feedstock containing phenols, neutral oils and/or tar bases comprises the

5 steps of

- i) subjecting the feedstock to liquid-liquid extraction using a solvent compound to transfer the phenols into a solvent layer and using a counter-solvent to transfer the neutral oils and/or nitrogen bases and at least some solvent into a counter-solvent layer;
- 10 ii) separating the solvent and counter-solvent layers with the counter-solvent layer containing some solvent compound;
- iii) recovering the counter-solvent from the counter-solvent layer to yield recovered counter-solvent as well as a mixture of solvent compound and neutral oils and/or tar bases;
- 15 iv) separating the solvent compound from the neutral oils and/or tar bases from the mixture of solvent and neutral oils and/or tar bases obtained in step iii); and
- v) recovering phenols from the solvent layer of step i) to yield a purified phenols product.

20

Recovery of the counter-solvent in step iii) may be by means of distillation, preferably by distillation under pressure. Preferably the counter-solvent is selected to be light boiling relative to the neutral oils and/or tar bases and thus

the counter-solvent goes to the overheads product during separation by distillation. The recovered counter-solvent may be recycled to the liquid-liquid extraction step.

- 5 The counter-solvent layer of step ii) will contain at least some solvent compound (usually due to the solubility of the solvent compound in the counter-solvent) and it will usually also contain some phenols. Although the amount of solvent in the separated counter-solvent layer of step ii) can be adjusted by adjusting operation conditions of the liquid-liquid extraction, such adjustments impact on
- 10 the effectiveness of the liquid-liquid extraction. The solvent (and possibly some of the phenols) in the separated counter-solvent layer will end up in the bottoms product with the neutral oils and/or tar bases if distillation is used to separate the counter-solvent from the counter-solvent layer. In the prior art such solvent and phenols were lost. However, this bottoms product is now treated to
- 15 separate the solvent and possibly some phenols from the neutral oils and/or tar bases.

In a preferred embodiment of the invention the separation of the solvent in step iv) is achieved by extracting the solvent from the mixture of solvent and neutral oil and/or tar bases by means of an extraction compound. The extraction compound may comprise a liquid, and preferably it comprises water. The water may comprise water not generated during the process; or water recycled from a process step of the process for purifying phenols

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from a feedstock. The extraction compound may be mixed with the mixture of solvent and neutral oils and/or tar bases obtained in step iii) to extract the solvent. This mixture may be subjected to phase separation to obtain an organic phase containing the neutral oils and/or nitrogen bases and an
5 aqueous phase containing the water and some solvent compound and usually also some phenols. The aqueous phase may then be recycled to the liquid-liquid extraction step, preferably by adding it to the solvent to be added to the liquid-liquid extraction step. The phase separation may be achieved by a liquid-liquid extraction unit, but preferably the mixture of
10 phases is allowed to settle causing phase separation, which phases are then separated, for example, decanted.

In a preferred embodiment of the invention the solvent compound used in step (i) may form part of a solvent in the form mixture of water and a
15 solvent compound, and at least some of the solvent compound being transferred to the counter-solvent layer while the rest of the solvent compound with at least some water is transferred into the solvent layer. In this scenario the phenols may be recovered from the solvent layer by separating the water from the solvent layer to yield recovered water as well
20 as a mixture of solvent compound and phenols; and thereafter separating the phenols from the solvent compound.

Preferably the water is recovered by subjecting the solvent layer to

- distillation, preferably distillation at or above atmospheric pressure. The distillation will yield a mixture comprising recovered water and some counter-solvent as an overheads product and a mixture comprising the solvent compound and phenols as a bottoms product. Preferably the
- 5 distillation of this step is operated at a pressure above 0 kPag , preferably from 0 to 100 Kpag, preferably at about 20kPag. The distillation of this step preferably carried out at a temperature at which the solvent compound and preferably also the counter-solvent will not decompose. In the case where triethylene glycol is used as a solvent the temperature may be from 150 to
- 10 207°C, preferably about 195°C. The distillation of this step is preferably also performed under such conditions in order that the water concentration is larger than 50wt% throughout the distillation process preferably greater than 70wt%. A distillation column may be used to perform the separation and preferably the water concentration is larger than 50wt%, preferably larger than 70wt%
- 15 throughout the distillation column, excluding the re-boiler. Operating the distillation of this step under such conditions will result in some water ending up in the bottoms product of the distillation. This water can be separated out, if required, during later processing steps.
- 20 In a preferred embodiment of the invention the separated solvent layer of step (ii) is mixed with counter-solvent for extracting neutral oils and/or tar bases therein during or before the distillation to separate water. Preferably the counter-solvent is added to the feed of the distillation to separate water, that is addition prior to the said distillation. Preferably the counter-solvent is added at

a mass ratio of 0 to 0.5 to the separated solvent layer fed to the distillation of step iii), preferably of a ratio of about 0.04.

- The counter-solvent and preferably also the solvent compound preferably
- 5 comprise organic compounds and preferably the overheads product mixture of the distillation step to separate water is phase separated to form an aqueous phase and an organic phase, which phases are then separated from each other. The phase separation may be achieved by condensation of the overheads product mixture of the distillation step in question, which condensate
- 10 is then settled to cause the phase separation to take place.

- The separated organic phase of the overheads product mixture of the distillation step to separate water is preferably recycled to the liquid-liquid extraction step. This organic phase will usually contain counter-solvent; neutral oils and/or tar
- 15 bases and some phenols. By recycling this phase to the liquid-liquid extraction step the phenols and counter-solvent contained therein are not lost. In the prior art the phenols were lost.

- The separated water phase of the overheads product mixture of the distillation step to separate water may be purged or recycled. Preferably at least some of this water is used as an extraction compound to achieve separation in step iv).

By operating the distillation to separate water under atmospheric or higher pressures, the disadvantages of vacuum distillation are avoided or at least

reduced. Such a vacuum distillation would cause major losses of counter-solvent to the vacuum system used to create the vacuum. It also improves control of the distillation, but it particularly causes improved separation of the organic and aqueous phases in the overheads product mixture (ie. no counter-solvent is required to be added after condensation of the overheads to step iii).

The addition of counter-solvent to the distillation of the step to separate water also improves recovery of phenols to the said organic phase. It further improves stripping of neutral oils and/or tar bases during the distillation of the step to separate water. Improved capital and operating costs are also achieved.

Recovery of phenols of step v) may be achieved by distillation, preferably distillation under vacuum. Water usually forms part of the bottoms product mixture of the water distillation step and at least some of this water ends up in the vacuum system, used to create a vacuum when the phenols are recovered under vacuum according to step v). The vacuum system is preferably operated in order to ensure that at least 50wt%, but preferably most of the water is lost to the vacuum system. This assists in drying the phenols.

20

However, phenols will also be lost to the vacuum system, but this loss can be minimized by recycling water with phenols therein recovered from the vacuum system, for example to the distillation to separate water. The water recovered from the vacuum system may be recycled to the feed stream to the distillation

to separate water.

The liquid-liquid extraction may comprise counter-current liquid-liquid extraction and a fractional liquid-liquid extraction may be used.

5

The solvent mixture may comprise a mixture of water and polar solvent, preferably a glycol. Preferably the glycol comprises triethylene glycol (TEG).

The mass ratio of water to glycol may be from 0.1 to 0.3 preferably it is about

0.2. The mass ratio of solvent mixture to feedstock may be from 0.3 to 3,

10 preferably about 0.7

The counter-solvent may comprise a non-polar compound, preferably it comprises a paraffinic compound or a mixture of paraffinic compounds (e.g. paraffins and isoparaffins). In one embodiment of the invention the counter-

15 solvent may comprise hexane. The mass ratio of counter-solvent to feedstock

may be from 0.5 to 3, preferably about 1.8.

Preferably the solvent compound is low boiling relative to the neutral oils and/or tar bases, and preferably the solvent is high boiling relative to the neutral oils

20 and/or tar bases.

The phase separation of step ii) may be achieved by any convenient manner e.g. decanting or during fractional extraction.

The invention also relates to phenols purified according to the process.

The invention will now be further described by means of the attached Figure which is a diagrammatic layout of a process according to the invention.

5

Referring now to the Figure a method of purifying phenols in a feedstock stream 10 containing phenols, neutral oils and tar bases comprises as a first step subjecting the feedstock stream 10 to liquid-liquid extraction (LLE) in a liquid-liquid extraction unit 11. The feedstock stream 10 was a concentrated phenols

10 mixed feed stream comprising 90 wt% phenols , the remainder being neutral oils and nitrogen bases in the naphtha/medium creosote range. This feedstock stream 10 was mixed with a feedstream of naphtha 12 having a phenolic content of ca. 31 wt% at a mass ratio of 0.3 to the feed stream 11. The concentrated phenolic stream is cleaned up in the process while the dilute 15 phenolic stream (feed stream 12, naphtha) allows for the production of additional phenolics.

Counter solvent in the form of hexane was fed via stream 13 to the bottom of the LLE unit 11 at a mass ratio of 1.8 to the combined feed streams 10 and 12 .

20 Other non-polar counter-solvents in the form of paraffinic compounds or mixtures thereof may be used in other applications. Solvent in the form of a mixture of water and a solvent compound in the form of triethylene glycol (TEG) in a mass ratio of 0.2 water/0.8 TEG. was fed via stream 14 to the LLE unit 11 at the top thereof. The solvent feed 14 was fed at a mass ratio of 0.7 to the

combined feeds 10 and 12. Other polar solvents, especially aqueous polar solvents may be used in other cases.

The LLE unit 11 can be any conventional LLE unit (e.g. packed, Karr, rotating disc contactor), but in this case it was a Scheibel type counter-current unit. The impeller diameter of the unit 11 was different throughout the length of the column to maximize mass transfer and throughput before flooding commences.

The LLE unit 11 may be operated at various temperature (e.g. 25 to 70°C) but in this particular example it was operated at a temperature of 45°C.

10

The raffinate stream 15 of the LLE unit 11 contained counter-solvent, and smaller amounts of phenols and TEG (due to its solubility in the counter-solvent). The raffinate stream 15 was fed to a distillation column (or rafinate stripper) 16 to recover the counter-solvent as an overheads product stream 17.

15 which was condensed in a condenser 18 and transferred to a reflux drum 19.

The recovered counter-solvent was recycled via stream 20 to the LLE unit 11.

Counter-solvent make up may be added via stream 21 to the reflux drum 19 as this gives a quick response to surges. Alternatively [not shown], counter-solvent

make-up may be added to the counter-solvent feed stream 13. The distillation

20 column 16 may be run at 0-200kPag pressure and a temperature profile from 55

to 230° C. In this example the pressure was 70kPag and the reboiler

temperature was 215°C.

The bottoms product stream 22 of the distillation column 16 was rich in neutral

oils and tar bases, but also included some TEG and some phenols. This bottoms product stream 22 was mixed with water at a ratio of 0.4, which water was from stream 23 (which will be described later). The resultant mixture stream 24 was fed to a decanter 25 where it phase separated into an organic layer stream 26 of neutral oils and tar bases which was further worked up [not shown]; and an aqueous layer stream 27 containing water and recovered TEG and some recovered phenols. The stream 27 was recycled to the LLE unit 11, particularly to the solvent feed stream 14. The one main advantage of addition of water via stream 23 and the separation in 25 is that TEG is recovered, which is then recycled. The use of water via stream 23 also minimizes the water balance (i.e. purging of water). In the decanter 25 the temperature was 55°C and the pressure was 15kPag, but other temperatures e.g. 25 to 75°C and other pressures may also be used.

15 The bottoms stream 28 from the LLE unit 11 mainly comprised solvent compound (TEG), water, phenols, some counter-solvent, and some neutral oils and tar bases. The stream 28 was fed to a distillation column or extract stripper 29 and it was mixed with a slipstream 30 of counter-solvent and a stream 34 waste water from a vacuum system, in particular steam ejectors (not shown, but described later) at a mass ratio of 0.04 counter solvent and 0.04 water from ejectors to total feed to extract stripper. The extract stripper 29 was operated under pressure, namely a pressure of 30 kpag. Some water was removed with the solvent compound (TEG) and phenols via the bottoms stream 32. The water dramatically decreased the reboiler [not shown] temperature which

prevented TEG degradation. Although most of the water was removed via the overheads stream 33 (which contained water, some counter-solvent [which was light boiling], some neutral oils and tar bases, and some azeotroped phenols) the unit 29 no longer operated as a pure water recovery unit but as an extract
5 stripper as it stripped the remaining neutral oils or tar bases from the phenols and the solvent compound.

The overheads stream 33 was condensed by means of the condenser 34 and the condensate was then phase separated in the reflux drum/decanter 35 into
10 an aqueous layer stream 36 and an organic layer stream 37. The aqueous layer stream 36 was essentially free of phenols, neutral oils and tar bases and was partly fed via stream 38 as reflux to the stripper 29. Some of the remaining water, that is in stream 39, was worked up and some of this was purged to close the water mass balance, while some water, that is in stream 23, was mixed with
15 the stream 22 as described above.

The organic layer stream 37 comprised counter-solvent, phenolics, neutral oils, tar bases and was recycled to the LLE unit 11, particularly to the naphtha feed stream 12. (Ratio ca. 0.2:1 (mass)). Alternatively it may be added to the
20 counter-solvent feed stream 13 or the solvent feed stream 14, but the latter is not suggested. Since the stripper 29 was operated under pressure measurable amounts of phenols were lost to the overheads stream 33, but it was not lost to the process as it was recycled to the LLE unit 11 via stream 37.

Addition of the counter-solvent via stream 30 to the bottoms stream 28 prior to the stripper 29 improved phase separation in the decanter 35. Running the stripper 29 under pressure allowed one to add counter-solvent to the stripper 29 without loss of the counter-solvent to a vacuum system (as in the prior art).

5 More phenols were removed overhead via stream 33 but it was recovered via the recycling of the organic layer stream 37. Pressure operation of the stripper 29 gave more control to the stripper 29 and allowed for improved operation of the reflux drum/decanter 35.

10 The bottoms stream 32 contained solvent compound (TEG), phenols and some water and was forwarded to a distillation column or product stripper 40 which operated under vacuum to strip the phenols as an overheads stream 41. The vacuum system [not shown] was run to ensure that at least 50 wt%, but preferably more water was lost to the vacuum system via stream 42. Dirty 15 solvent product was recovered as the bottoms stream 43. The unit 40 was operated under vacuum because of the solvent compound (TEG) degradation temperature and the stream saturation temperature. Typically the operating pressure is 3,5kPa abs and typically the bottoms temperature is <195°C, usually about 195.°C.

20

The dirty solvent stream 43 was recycled back to LLE unit 11 especially to the solvent the solvent feedstream 14. A small slip stream 44 was cleaned by means of flash evaporation in the unit 45. The clean solvent stream 46 was mixed with the dirty solvent stream 43. The bottoms product stream 47 was

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removed to be processed further. The flash evaporation 45 was typically run at 2kPa abs and at a temperature of 167°C.

The vent 48 of the flash was recovered in the vacuum system [not shown] which
5 was recovered as a waste water stream 31, which was returned to the stripper
29.

The addition of the waste water stream 31 from the vacuum system of unit 40
means that phenols lost to the vacuum system may be recovered in the stripper
10 29. The waste water stream 31 may be added to the reflux drum/decanter 35
but it is not recommended as it may contain solvent which will be purged via the
stream 39.

Make up TEG can be added as stream 49.

15 The separated phenols of stream 41 had a composition of 95%+ phenols, and
ca. 1.6 wt% water. If required the water may be removed in a subsequent step
by means of for example a distillation unit.

20 It will be appreciated that many variations in detail are possible without thereby
departing from the scope and spirit of the invention.

Dated this

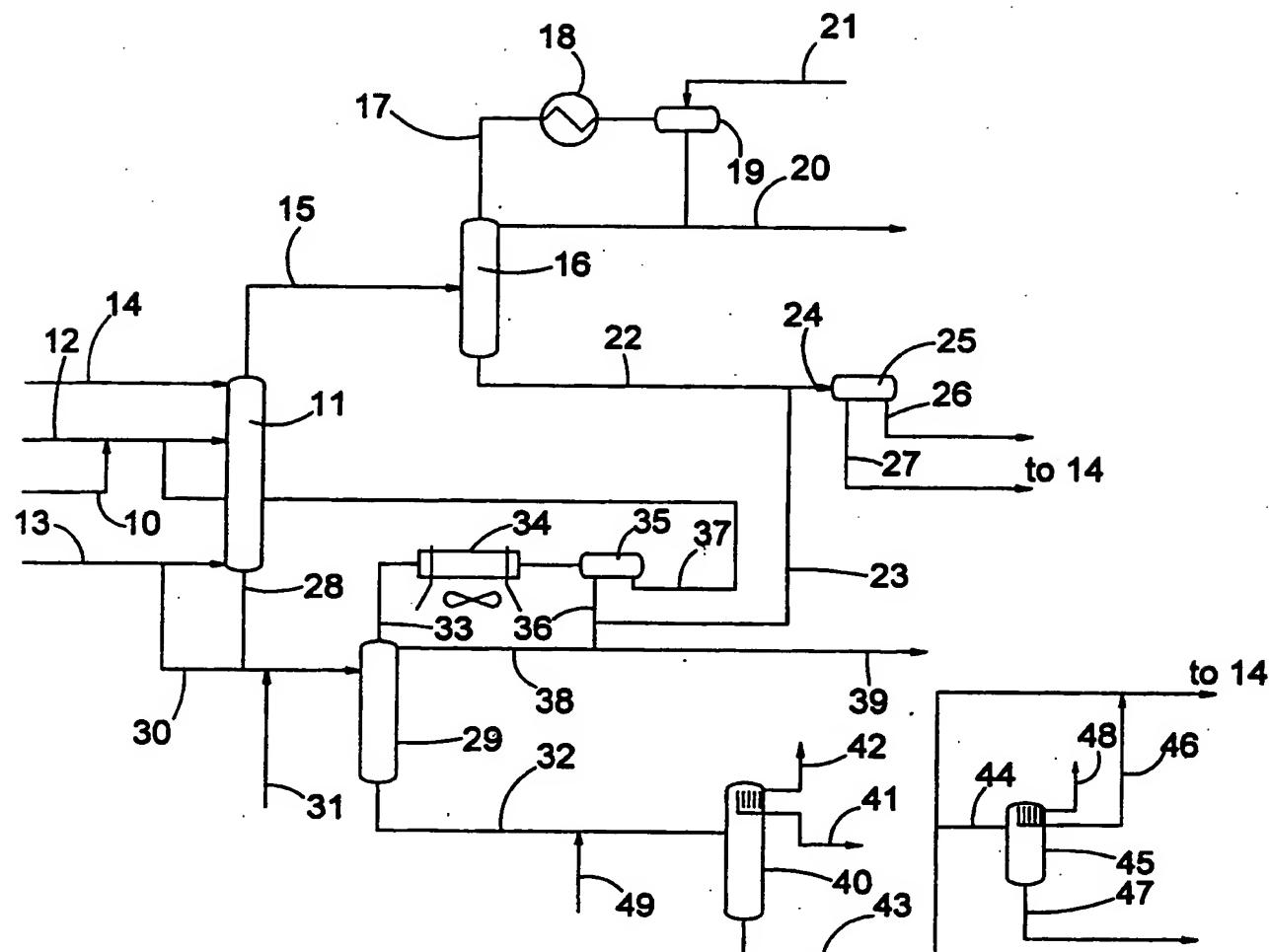
3 day of April 2003

Patent Attorney Agent for the Applicant

2003/2621

MERISOL (RSA) PTY LTD

1 SHEET



FIGURE